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Effect of residual compressive surface stress on severe wear of alumina–silicon carbide two-layered composites

C.E.J. Dancer [a,b,*], N.A. Yahya [a,c], T. Berndt [a,d], R.I. Todd [a], G. de Portu [a,e]

[a] Department of Materials, University of Oxford, Parks Road, Oxford, OX1 3PH, UK
[b] Warwick Manufacturing Group, University of Warwick, Coventry, CV4 7AL, UK
[c] Faculty of Dentistry, University of Malaya, Kuala Lumpur, 50603, Malaysia
[d] Faculty of Mechanical Engineering, Otto-von-Guericke-Universität Magdeburg, Universitätsplatz 2, D-39016 Magdeburg, Germany
[e] Research Institute of Science and Technology for Ceramics, ISTEC-CNR, Via Granarolo 64, 48018 Faenza, Italy

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ABSTRACT

Ceramics consisting of Al_2O_3 with a surface layer of Al_2O_3-10 vol% SiC have been fabricated by hot pressing. The residual compressive stress at the composite surface due to the difference in thermal expansion between the two layers has been measured experimentally by Cr^{3+} fluorescence microspectroscopy. The wear resistance in the severe wear regime of the two-layered samples was higher than those of a reference single-layer Al_2O_3-10 vol% SiC sample. The improvement in the wear resistance was due to a decrease in the amount of surface pullout which was attributed to the presence of the biaxial residual compressive stress in the surface layer of the specimens.

1. Introduction

Ceramics are suitable for many tribological applications because they have high stiffness, high hardness, low surface friction and are generally corrosion resistant [1,2]. However as the wear mechanism in ceramics such as Al_2O_3 can involve brittle fracture leading to grain pullout and/or cracking in severe conditions [3], it is apparent that the wear resistance can be improved by increasing the resistance of the ceramic material to surface fracture [2,4,5]. One way of doing this is to induce compressive stresses in the surface of the ceramic. This can be achieved by methods including lamination (creating ceramic materials from thin layers of two or more compositions with different coefficients of thermal expansion (CTE)) [4,6,7] and thermochemical treatments which induce phase changes [8,9]. For the Al_2O_3-ZrO_2 system laminated samples made from stacks of tape-cast materials sintered together have been shown to have improved wear resistance [1,5] and greater apparent toughness [5,10,11] compared to monolithic Al_2O_3 or ZrO_2 samples. The compressive residual stresses in the surface of the sample have been measured by methods including the indentation technique [5,10] and piezospectroscopic microscopy [1]. By comparing the magnitude of the compressive residual stress with the wear resistance, de Portu et al. [1] found that Al_2O_3–ZrO_2-based structures with greater compressive stresses have superior wear performance. The authors compared the results of abrasive wear tests performed on pure Al_2O_3 with those of Al_2O_3/Al_2O_3–ZrO_2 layered composites which exhibited compressive residual stresses on the outer Al_2O_3 layer. They found that the wear resistance $1/k$ was 3.3×10^{13} Nm^{-2} for stress-free Al_2O_3 and 3.6×10^{13} Nm^{-2}, 4.0×10^{13} Nm^{-2} and 7.7×10^{13} Nm^{-2} for the layered structures with compressive residual stresses of −27 MPa, −45 MPa and −166 MPa, respectively [1]. The apparent fracture toughness has been measured by the indentation method [5,12,13] and by the single-edge V-notch beam method [11] and has been shown to vary with respect to the stress field such that in regions with greater compressive stresses the apparent fracture toughness is higher. However lamination involves more complicated processing routes compared to monolithic ceramics, as the layers must first be tape cast, then stacked, and finally sintered [4].

Al_2O_3–SiC "nanocomposite" materials have demonstrably better wear resistance compared to monolithic Al_2O_3 [2,14–20]. The addition of SiC to Al_2O_3 causes a change in the fracture mode from intergranular to transgranular [21–23]. This reduces the size of the fragments of surface removed by fracture and pullout [16]. The change in fracture mode has also been shown to enhance the toughness in alumina–silicon carbide whisker nanocomposites [24]. Any increase in toughness will particularly affect the short surface cracks important in severe wear, as the crack deflection-induced bridging in intergranular fracture, which can compensate for the low toughness of the grain boundaries, and is only

* Corresponding author at: Warwick Manufacturing Group, University of Warwick, Coventry, CV4 7AL, UK.
E-mail address: c.dancer@warwick.ac.uk (C.E.J. Dancer).

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activated after the crack has travelled a distance greater than the grain size. In addition, the presence of intragranular SiC particles is thought to lead to the suppression of the twinning and dislocation activities responsible for crack initiation [15]. For Al_2O_3-5 vol% SiC samples polished with different treatments, Wu et al. [25] observed the presence of plastically deformed regions at the polished surface by transmission electron microscopy and observed more plastic deformation in the nanocomposites. Hertzian indentation measurements of the residual stress determined that a higher residual compressive stress was present in the surface of the nanocomposite samples compared to the equivalently polished Al_2O_3 [25]. In a later study, Guo et al. [26] showed that the main reason for the higher mean compressive stress in the nanocomposites was the retention of plastically deformed grains owing to the suppression of fracture and pullout. This also results in a greater uniformity of compressive stress and avoids the occurrence of tensile stress.

These properties of Al_2O_3–SiC nanocomposites lead to a reduction in both the number and average size of pullouts in the composite compared to the Al_2O_3, and hence a reduction in the wear rate [15,16]. This is readily illustrated by the observation that Al_2O_3–SiC nanocomposites can be polished much more easily than monolithic Al_2O_3 of a similar grain size [27,28].

Both these approaches – controlling surface compressive stresses due to CTE mismatch in layered structures, and the production of Al_2O_3–SiC nanocomposites – result in improved wear resistance due to the suppression of cracking. However to the best knowledge of the authors combining these two approaches in a single structure in order to improve the wear resistance has not previously been attempted. This paper examines the effect of compressive residual stress on the wear properties and cracking behaviour of Al_2O_3-10 vol% SiC materials by comparing "monolithic" Al_2O_3-10 vol% SiC samples with two-layer samples consisting of Al_2O_3 faced with a layer of Al_2O_3-10 vol% SiC nanocomposite. The latter were produced using methods previously developed to produce functionally graded Al_2O_3–SiC materials [29]. As the monolithic and layered structures are manufactured using identical processing methods, this has the potential to be a straightforward route to improve further the already excellent wear properties of Al_2O_3–SiC nanocomposites.

2. Experimental

2.1. Sample fabrication

Al_2O_3 (AES11c, Sumitomo; 400 nm particle size) and 10 vol% SiC (UF-25, HC Starck; 450 nm particle size) powders were mixed with distilled water and 2.1 g of a dispersing agent (Dispex A40, Allied Colloids). This mixture was attrition milled (Union Process) for 2 h at 300 rpm using a ZrO_2 tank and blade and 3 mm diameter Y_2O_3-stabilized ZrO_2 balls. Following milling, the powder was freeze-dried (Edwards MultiModulyo) and passed through a 150 μm aperture sieve. The powder was then calcined at 600 °C for 2 h to remove the organic dispersant.

Samples were hot pressed in a graphite die of 25 mm diameter. Two-layered composites were assembled by first filling the die with an appropriate quantity of Al_2O_3 powder which was lightly tamped down flat before adding the appropriate quantity of the Al_2O_3-10 vol% SiC powder such that the layer thicknesses in the as-pressed specimens would be approximately 4 mm of Al_2O_3 and 2 mm of Al_2O_3-10 vol% SiC. Graphite foil and graphite spacers were placed between each sample in the die. A monolithic reference Al_2O_3-10 vol% SiC sample (AS) and the two-layered samples (TL1 and TL2) were hot pressed simultaneously in argon at 1650 °C for 30 min under 25 MPa uniaxial pressure, using a heating rate of 15–30 °C/min and an initial cooling rate of 15 °C/min, followed by furnace cooling.

Following hot pressing the samples were removed from the graphite die and ground on a flatbed grinder on both sides to remove residual carbon and to ensure a flat surface. The Al_2O_3-10 vol% SiC surfaces were then sequentially polished to a 1 μm finish using diamond pastes. This ensured that the stresses determined from the samples are representative of the sample rather than being due to grinding stresses, and minimised the noise in the Cr^{3+} fluorescence spectra.

The bulk density of the samples was measured using the Archimedes method with distilled water as the immersion medium. The average grain size in both layers of each laminate and for the reference sample were measured using the linear intercept method with a multiplication factor of 1.56 [30] from scanning electron microscopy (SEM) images (JEOL 6500 F) of polished and thermally-etched (in vacuum at 1450 °C for 30 min) cross-sections cut from each sample using a diamond blade. Samples were coated with 2 nm of platinum prior to examination in the SEM.

Vickers indentations were made using a 1 kg load and the size of the indentations was measured using an Axioskop 2 MAT optical microscope (Zeiss) and AxioVision software (v.4.8, Imaging Associates/Zeiss) in order to determine the hardness. In addition larger indentations were made using loads of 5, 10 and 15 kg and the lengths of cracks originating at the sharp corners of these Vickers indentations were measured by optical microscopy. As the crack length was observed to increase with time, for comparison all crack lengths were measured exactly 15 min after the indentation was made. The increase in crack length with time was determined for 5 kg indentations only. At least 6 indentations were made in each sample.

2.2. Surface stress measurements

To measure the change in surface stress due to the layering, a piezospectroscopic technique was used which relates the R1 Cr^{3+} fluorescence peak position to the stress in the sample. The biaxial stress state in these samples is due solely to the difference in composition between the layers which causes a difference in the coefficient of thermal expansion (CTE) between the layers. In the case of the samples studied in this work, the CTE of Al_2O_3 is $8.9\times10^{-6}\,°C^{-1}$ and that of the Al_2O_3-10 vol% SiC nanocomposite material is $8.45\times10^{-6}\,°C^{-1}$. Therefore the relationship between the change in frequency of the R1 peak $\Delta\nu$ and the biaxial surface stress σ takes the following form:

$$\sigma = \frac{3}{2\Pi_H}\Delta\nu \quad (1)$$

The hydrostatic piezospectroscopic coefficient Π_H is $7.59\,cm^{-1}\,GPa^{-1}$ for polycrystalline texture-free Al_2O_3 [31].

Cr^{3+} fluorescence spectra were recorded from 5 regions over the surface of each sample using a 1000 series Renishaw Raman microscope (Renishaw, UK) with a 50 mW He–Ne laser and an automated X–Y–Z stage (Prior, UK). Spectra were taken from points at least 4 mm from the sample edges to avoid edge related effects. All measurements were taken with confocal settings using a 100× objective lens. Spectra were curve-fitted using the WiRE software package (version 3.2, Renishaw, UK) and average values of the R1 peak position were calculated for each sample. The change in stress due to the lamination was then calculated from Eq. (1) by subtracting the R1 peak position for the two layer specimens from that for the reference sample of nanocomposite.

2.3. Wear tests

A micro-scale abrasive wear tester (TE 66, Phoenix Tribology, UK) was used in this study. A pre-conditioned chrome steel ball,